

A New 3D Coordination Polymer Based on Pentanuclear Cd(II) Rod-Shaped Secondary Building Unit: Synthesis, Crystal Structure and Luminescent Property

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Abstract A new coordination polymer $[\text{Cd}_{2.5}(\mu_3\text{-OH})(\text{L})_2(\text{bipy})]_n$ (**1**). (L = 1,2-naphthalene dicarboxylate, bipy = 4,4'-bipyridine), was obtained under hydrothermal condition. The structure of **1** has been determined by single-crystal X-ray diffraction analysis and further characterized by elemental analysis and IR spectrum. Single-crystal X-ray diffraction analyses reveal that complex **1** crystallizes in triclinic system, $P\bar{1}$ space group. Complex **1** possesses a 3D pcu net based on a pentanuclear Cd(II) cluster. Each pentanuclear Cd(II) cluster links with neighboring ones and generates a rod-shaped secondary building unit, which is further extended by bipy ligands to forms a three-dimensional network. Furthermore, the luminescent properties of this complex and the free ligand have been investigated. Complex **1** can be excited directly and emit strong solid-state fluorescence at room temperature, which may make them excellent candidates for fluorescent materials.

Keywords Complex · Pentanuclear cluster · Secondary building unit · Luminescent properties

1 Introduction

During the past two decades, metal–organic frameworks (MOFs), as an important class of crystalline solid materials, have received tremendous attention because of their potential in various applications, such as gas adsorption

and separation, catalysis, drug delivery, electrochemistry, molecular recognition, sensing, and detection [1–13]. The major task of the synthesis for such MOFs is to choose appropriate metal-connecting nodes and organic bridging ligands to control the formation of these complexes with required structures and properties. Among this topic realm, as an attractive subarea, homometallic cluster chemistry has been a small but growing family in recent years. In this case, homometallic cluster as secondary building units (SBUs) have been proved as an effective and powerful synthetic strategy in constructing new MOFs [14]. Depending on their architecture, MOFs based on polynuclear metallic units offer potential for unique properties, such as luminescence, magnetism and catalysis [15].

When the coordination number and geometry of metal centers are relatively fixed, organic ligands acting as bridging linkers to connect metal centers play the essential role [16]. From the point of view of organic ligand design, it is desirable to synthesize bulky ligands that have the coordinating atoms pointing on one side of the molecule, this favoring isolation of diverse discrete (dinuclear, tetranuclear, pentanuclear, or hexanuclear) and infinite (1D, 2D, or 3D) structures [17]. Following this strategy, the aromatic chelate ligand phthalic acid has been used as a good ligand for the construction of coordination polymers with polynuclear clusters due to the ability to bind more metals and isolate discrete metal cluster. For example, one decanuclear manganese [18] and three uranyl [19] compounds have been reported by G. Christou, in the presence of phthalate. To investigate the influence of bulky aromatic skeletons of such ligands on structures and properties of their coordination complexes, the naphthalene-based dicarboxyl ligand 1,2-naphthalene dicarboxylic acid (H_2L), an expansion of phthalic acid, was chosen as the organic ligand in our recently work. To date, coordination

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polymers constructed from the **L** ligand have rarely been synthesized. Also, the introduction of naphthalene based ligands into coordination polymers might lead to interesting luminescence properties of the final product. In this paper, we report the syntheses and characterizations of one coordination polymers $[\text{Cd}_{2.5}(\mu_3\text{-OH})(\text{L})_2(\text{bipy})]_n$ (**1**) based on 1,2-naphthalene dicarboxylic acid and 4,4'-bipyridine. To our interest, this complex consists of pentanuclear Cd(II) rod-shaped SBUs and exhibits interesting luminescent properties.

2 Experimental Section

2.1 General Procedures

All chemicals and reagents were used as received from commercial sources without further purification. All reactions were carried out under hydrothermal conditions. Elemental analyses (C, H, N) were determined with a Elementar Vario EL III elemental analyzer; IR spectra were recorded as KBr pellets on a Bruker EQUINOX55 spectrophotometer in the 4000–400 cm^{-1} region. Fluorescence spectra were performed on a Hitachi F-4500 fluorescence spectrophotometer at room temperature. The X-ray powder diffraction pattern was recorded with a Rigaku D/Max 3III diffractometer.

2.2 Synthesis of Complex



A mixture of H_2L (10.8 mg, 0.05 mmol), $\text{Cd}(\text{OAc})_2 \cdot 2\text{H}_2\text{O}$ (26.6 mg, 0.1 mmol), bipy (15.6 mg, 0.1 mmol) and KOH (5.6 mg, 0.1 mmol) were added to water (12 mL) in a 25 mL Teflon-lined stainless steel vessel. The mixture was heated at 180 °C for 4 days. After the reactive mixture was slowly cooled to room temperature, colorless block crystals of **1** were obtained (yield: 50 %, based on Cd). Anal. Calc. (%) for $\text{C}_{34}\text{H}_{20}\text{Cd}_{2.5}\text{N}_2\text{O}_9$: C 46.32, H 2.29, N 3.18; found (%): C 46.19, H 2.12, N 3.05. IR (KBr pellet, cm^{-1}): 3390(w, br), 2361(w), 1597(s), 1562(vs), 1507 (m), 1465(m), 1407(s), 1383(s), 1314(w), 1265(w), 1218(w), 1146(w), 1060(m), 1033(m), 938(m), 868(w), 796(m), 773(m), 746(w), 664(w), 625(w), 589(w).

2.3 Crystal Structural Determination

Diffraction intensities for the complex **1** was collected at 294(2) K on a Bruker SMART 1000 CCD diffractometer employing graphite-monochromated Mo- $K\alpha$ radiation ($\lambda = 0.071073$ nm). A semiempirical absorption correction was applied using the SADABS program [20]. The structure was solved by direct methods and refined by full-

Table 1 Crystallographic data and structure refinement parameters for complex **1**

Complex	1
Empirical formula	$\text{C}_{34}\text{H}_{20}\text{N}_2\text{O}_9\text{Cd}_{2.5}$
Formula weight	881.52
Crystal system	Triclinic
Space group	$P\bar{1}$
$a/\text{\AA}$	7.7878(9)
$b/\text{\AA}$	13.8854(13)
$c/\text{\AA}$	15.0832(14)
$\alpha/^\circ$	64.539(9)
$\beta/^\circ$	79.268(9)
$\gamma/^\circ$	87.711(9)
Volume/ \AA^3	1445.4(3)
Z	2
$D/\text{g cm}^{-3}$	2.028
μ/mm^{-1}	1.894
$F(000)$	862
R_{int}	0.0704
T/K	294(2)
Goodness-of-fit on F^2	0.990
R_1^a/wR_2^b [$I > 2\sigma(I)$]	0.0455/0.0807
R_1^a/wR_2^b (all data)	0.0827/0.0929
$\rho_{\text{max}}/\rho_{\text{min}}$ (e \AA^{-3})	0.893/−0.868

$$^a R_1 = \Sigma(|F_o| - |F_c|)/\Sigma|F_o|$$

$$^b wR_2 = [\Sigma w(|F_o|^2 - |F_c|^2)^2/\Sigma w(F_o^2)^2]^{1/2}$$

matrix least-squares on F^2 using the SHELXS 97 and SHELXL 97 programs, respectively [21, 22]. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were placed in geometrically calculated positions. Crystal data and structural refinement parameters for **1** are summarized in Table 1. Selected bond distances and bond angles are listed in Table 2. Crystallographic data (excluding structure factors) for the structures in this paper have been deposited with the Cambridge Crystallographic Data Centre, CCDC, 12 Union Road, Cambridge CB21EZ, UK. Copies of the data can be obtained free of charge on quoting the depository number CCDC-1006522 for **1** (Fax: +44-1223-336-033; E-Mail: deposit@ccdc.cam.ac.uk, <http://www.ccdc.cam.ac.uk>).

3 Results and Discussion

3.1 Structure Description of $[\text{Cd}_{2.5}(\mu_3\text{-OH})(\text{L})_2(\text{bipy})]_n$ (**1**)

Single-crystal X-ray diffraction analyses reveal that complex **1** crystallizes in triclinic system, $P\bar{1}$ space group and exhibits a 3D coordination framework constructed from a

Table 2 Selected bond lengths (Å) and angles (°) for complex **1**

Cd(1)–O(9)	2.224(4)	Cd(1)–O(7)#1	2.284(4)
Cd(1)–N(1)	2.313(5)	Cd(1)–O(1)	2.320(4)
Cd(1)–O(6)	2.355(5)	Cd(1)–O(8)#1	2.598(5)
Cd(2)–O(4)#2	2.204(5)	Cd(2)–O(7)	2.243(4)
Cd(2)–O(9)#3	2.283(4)	Cd(2)–O(2)#3	2.289(4)
Cd(2)–O(5)	2.411(4)	Cd(2)–N(2)	2.428(6)
Cd(3)–O(9)#3	2.235(4)	Cd(3)–O(9)	2.235(4)
Cd(3)–O(3)#4	2.260(4)	Cd(3)–O(3)#2	2.260(4)
Cd(3)–O(5)#3	2.322(4)	Cd(3)–O(5)	2.322(4)
O(9)–Cd(1)–O(7)#1	99.31(15)	O(9)–Cd(1)–N(1)	112.28(17)
O(7)#1–Cd(1)–N(1)	146.70(17)	O(9)–Cd(1)–O(1)	99.37(16)
O(7)#1–Cd(1)–O(1)	98.66(16)	N(1)–Cd(1)–O(1)	86.74(18)
O(9)–Cd(1)–O(6)	89.98(15)	O(7)#1–Cd(1)–O(6)	85.65(16)
N(1)–Cd(1)–O(6)	84.12(19)	O(1)–Cd(1)–O(6)	168.88(17)
O(9)–Cd(1)–O(8)#1	151.07(15)	O(7)#1–Cd(1)–O(8)#1	52.90(15)
N(1)–Cd(1)–O(8)#1	96.59(17)	O(1)–Cd(1)–O(8)#1	79.74(16)
O(6)–Cd(1)–O(8)#1	95.00(16)	O(4)#2–Cd(2)–O(7)	150.77(18)
O(4)#2–Cd(2)–O(9)#3	98.24(18)	O(7)–Cd(2)–O(9)#3	101.92(15)
O(4)#2–Cd(2)–O(2)#3	119.29(18)	O(7)–Cd(2)–O(2)#3	85.25(15)
O(9)#3–Cd(2)–O(2)#3	79.69(15)	O(4)#2–Cd(2)–O(5)	86.42(18)
O(7)–Cd(2)–O(5)	77.82(16)	O(7)–Cd(2)–Cd(3)	106.54(12)
O(9)#3–Cd(2)–O(5)	77.06(14)	O(9)#3–Cd(2)–Cd(3)	40.47(9)
O(2)#3–Cd(2)–O(5)	147.55(15)	O(2)#3–Cd(2)–Cd(3)	120.03(12)
O(4)#2–Cd(2)–N(2)	82.3(2)	O(5)–Cd(2)–Cd(3)	42.88(10)
O(7)–Cd(2)–N(2)	86.35(18)	N(2)–Cd(2)–Cd(3)	154.94(13)
O(9)#3–Cd(2)–N(2)	158.84(16)	O(9)#3–Cd(3)–O(9)	180.0(2)
O(2)#3–Cd(2)–N(2)	81.67(17)	O(9)#3–Cd(3)–O(3)#4	91.33(15)
O(5)–Cd(2)–N(2)	123.97(16)	O(9)–Cd(3)–O(3)#4	88.67(15)
O(4)#2–Cd(2)–Cd(3)	76.14(14)	O(9)#3–Cd(3)–O(3)#2	88.67(15)
O(9)–Cd(3)–O(3)#2	91.33(15)	O(3)#4–Cd(3)–O(3)#2	180.00(11)
O(9)#3–Cd(3)–O(5)#3	100.12(14)	O(9)#3–Cd(3)–O(5)	79.88(14)
O(9)–Cd(3)–O(5)#3	79.88(14)	O(9)–Cd(3)–O(5)	100.12(14)
O(3)#4–Cd(3)–O(5)#3	91.11(17)	O(3)#4–Cd(3)–O(5)	88.89(17)
O(3)#2–Cd(3)–O(5)#3	88.89(17)	O(3)#2–Cd(3)–O(5)	91.11(17)

Symmetry codes for **1**: #1 $x-1$, y, z ; #2 $-x+1, -y, -z$; #3 $-x+2, -y, -z$; #4 $x+1, y, z$

planar pentanuclear Cd(II) cluster. The asymmetric unit consists of two and a half crystallographically unique Cd(II) centers, one μ_3 -hydroxyl group, two fully deprotonated **L** ligands, and one bipy ligand. As shown in Fig. 1a, Cd1 and Cd2 atom is coordinated by one nitrogen atom from one bipy ligand [Cd1–N1 = 2.313(5), Cd2–N2 = 2.427(6) Å], four carboxylate oxygen atoms from three **L** ligands [Cd1–O: 2.284(4)–2.599(5), Cd2–O: 2.203(5)–2.411(4) Å], and one hydroxyl oxygen atom [Cd1–O9 = 2.233(4), Cd2–O9C = 2.287(4) Å] to finish distorted octahedral geometry. The Cd3 atom is six-coordinated, adopting a distorted octahedral geometry via coordinating to four carboxylate oxygen atoms from four **L** ligands [Cd3–O: 2.261(4)–2.323(4) Å], and two

hydroxyl oxygen atoms [Cd3–O9/O9C = 2.239(4) Å]. All the Cd–O [2.203(5)–2.599(5) Å] and Cd–N [2.313(5) and 2.427(6) Å] bond distances as well as the bond angles around each Cd(II) atom [52.94(14)–180.0°] are in the normal range [23, 24]. As shown in Fig. 1b, there are two kinds of **L** ligands in **1**. One adopts a μ_4 -bridging mode linking four Cd(II) atoms through two bidentate carboxylate groups. The other **L** ligand shows a μ_6 -bridging mode linking four Cd(II) atoms through one bidentate and one chelate-monodentate carboxylate groups.

The pentanuclear Cd(II) cluster (Fig. 2a) is composed of parallelogram arrangement of four Cd(II) atoms [Cd1, Cd2, Cd1A, Cd2A ($A = 2 - x, -y, -z$)] and a centered fifth Cd3 atom. The parallelogram shows two sides of 3.909

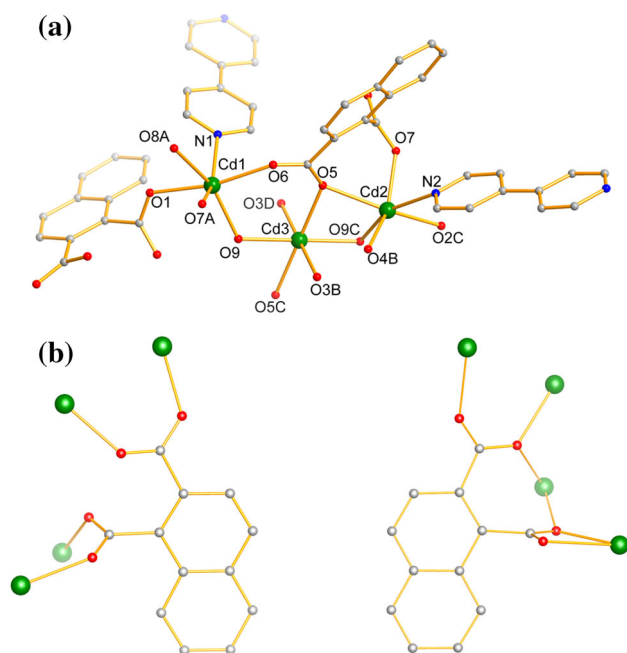
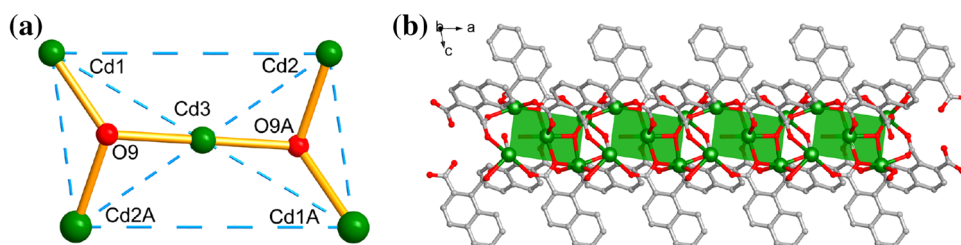


Fig. 1 Ball-and-stick views of the coordination geometries of Cd(II) in **1** (a) and (b) coordination modes of **L** ligand. Color scheme: gray for C, blue for N, red for O, and green for Cd

(Cd1...Cd2A) and 6.174 Å (Cd1...Cd2), two diagonals of 6.819 Å (Cd2...Cd2A) and 7.766 Å (Cd1...Cd1A), and two inner angles of 81.77 and 98.23°, respectively. The central Cd3 lies on a crystallographic inversion center, and therefore, the five Cd(II) ions are coplanar. The Cd(II) ions at each short edge of the rectangle are bridged by two hydroxyl oxygen atoms O9 and O9A. The central μ_3 -oxygen atom O9/O9A is displaced by 0.590 Å over the trigonal plane of the three Cd(II) atoms, and the Cd–O–Cd angles are 97.98, 120.30, and 121.15° respectively. In the coordination modes mentioned above, each pentanuclear Cd(II) cluster links with two neighboring ones and constructs a rod-shaped SBU (Fig. 2b), which is further linked to neighboring rod-shaped SBUs with bipy ligands and thus give rise to a 3D network (Fig. 3). The structure of complex **1** has rhombic channels of 20.9×8.7 Å dimensions with a void space running along *a* direction and the channels are occupied by naphthalene motif of **L** ligands.

Fig. 2 Ball-and-stick view of the pentanuclear Cd(II) subunit (a) and (b) infinite rod-shaped secondary building block



Up to now, some reports on MOFs constructed from rod-shaped SBUs have been reported [25–27], but rare examples possess high nuclear rod-shaped SBUs. To our knowledge, only one MOF based on octanuclear Cd(II) rod-shaped SBUs have been synthesized by Wang and co-workers [28]. We also found one copper(II) complex based on the similar pentanuclear cluster as that in **1** has been reported [29]. However, the 1,2-bis(imidazol-1-yl)ethane bridge extends pentanuclear Cu(II) rod-shaped SBUs resulting in a 2D layer but not 3D network. To fully understand the structure of **1**, the topological approach is applied to simplify such a 3D coordination framework. If each pentanuclear cluster is considered as one node, it connects six ones through two pairs of O7–C14–O8 carboxylate groups and four bipy ligands to generate a 6-connected pcu network (Fig. 4).

3.2 PXRD Results and Luminescent Property

In order to check the phase purity of the complex, powder X-ray diffraction (PXRD) patterns of **1** was recorded at room temperature. As shown in Fig. 5, the peak positions of the simulated and experimental PXRD patterns are in agreement with each other, which confirm its phase purity. The difference in intensity of some diffraction peaks may be attributed to the preferred orientation of the crystalline powder samples.

The luminescent properties of compounds containing d^{10} metal centers have been attracting more interest because of their potential applications in chemical sensors, photochemistry and electroluminescent display [30]. To examine the luminescent properties of the d^{10} metal complex, the luminescence spectra of **1** (Fig. 6) has been measured. Complex **1** displays emission peak at 410 nm upon excitation at $\lambda = 275$ nm. In order to understand the nature of such emission bands, the fluorescence properties of the free H_2L ligand was also measured, upon excitation at ca. 275 nm, which shows the emission at 455 nm. Compared with the emission spectrum of H_2L , the maximum wavelength of **1** is blue shifted by 45 nm, and the intensity is enhanced. We can attribute the blue-shift and the increase of emission intensity to the ligand chelation to the metal center, which effectively increases the rigidity

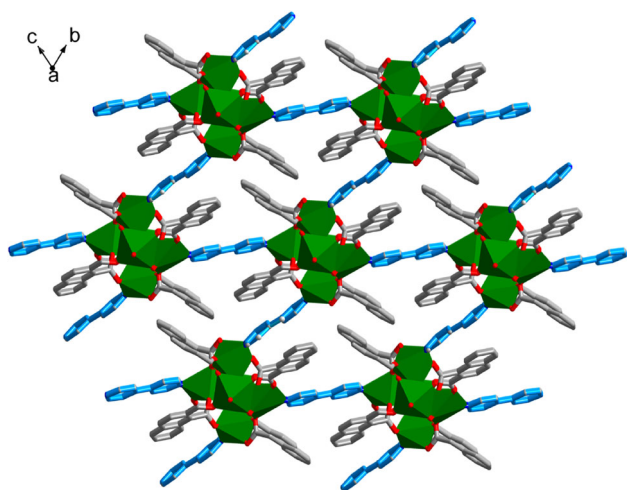


Fig. 3 Polyhedron representation of the 3D network of **1** along *a* direction, the bipy ligands are highlighted in *blue* for clarity

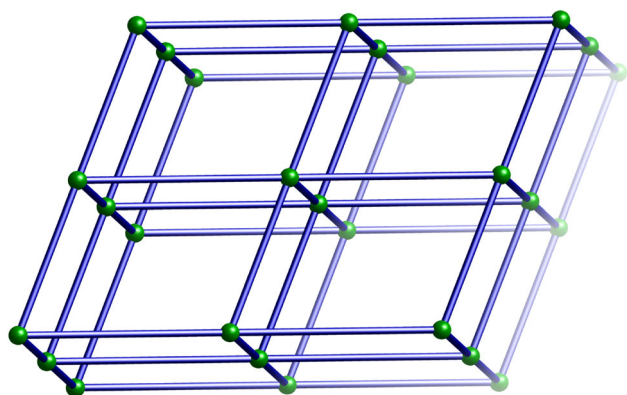


Fig. 4 Schematic view of the pcu net (green sphere for pentanuclear subunits)

and asymmetry of the ligand and reduces the loss of energy by radiation decay [31].

3.3 IR Spectra Analysis

As shown in Fig. 7, in the IR spectra of **1**, the broad bands centered at 3390 cm^{-1} indicates O–H stretching of hydroxyl group in **L**. As a matter of fact, the IR absorption of carboxylate group is very complicated due to its coordination diversities with metal ions. The characteristic bands of carboxylate groups in **1** appeared in the usual region at $1597\text{--}1507\text{ cm}^{-1}$ for the antisymmetric stretching vibrations and at $1465\text{--}1383\text{ cm}^{-1}$ for the symmetric stretching vibrations. Furthermore, the $\Delta\nu$ values [$\Delta\nu = \nu_{\text{asym}}(\text{COO}^-) - \nu_{\text{sym}}(\text{COO}^-)$] are 155 and 97 cm^{-1}

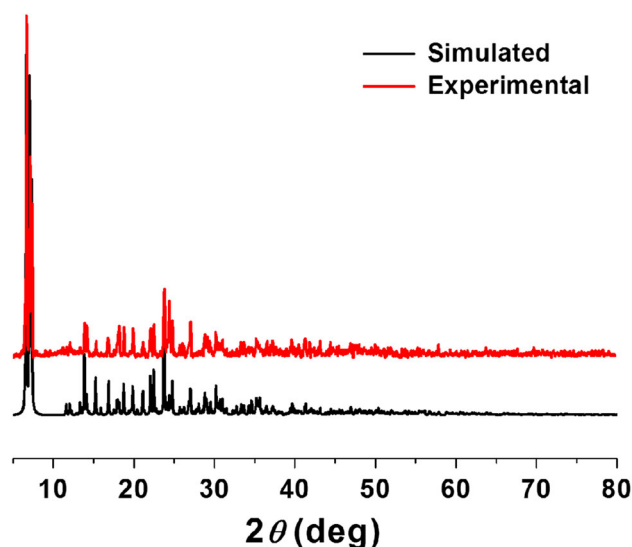


Fig. 5 XRD patterns of complex **1**

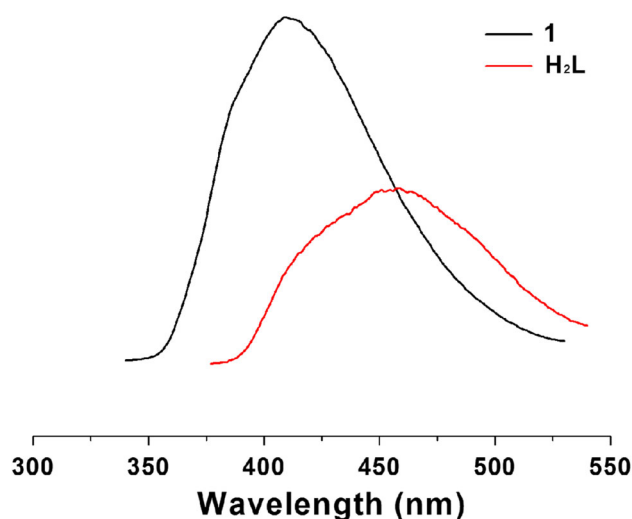


Fig. 6 Solid-state emission spectra of complexes **1** and **H₂L** ligand at room temperature

for **1**, which are in good agreement with their solid structural features from the results of their crystal structures.

4 Conclusion

In summary, a novel coordination polymer based on the naphthalene-based di-carboxyl ligand 1,2-naphthalene dicarboxylic acid (**H₂L**) has been synthesized under hydrothermal conditions. In the structure of **1**, the **L** ligand shows μ_4 - and μ_6 -bridging mode binding more Cd(II) atoms to form a parallelogram pentanuclear Cd(II) cluster,

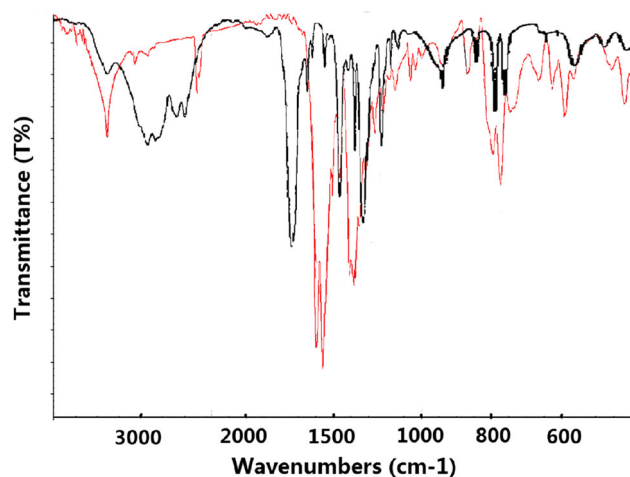


Fig. 7 FT-IR spectras of the complex **1** (red) and H_2L ligand (black)

then to a 1D rod-shaped SBU with the naphthalene motif of **L** ligand decorating on four sides. The bridging bipy ligands extended each rod-shaped SBU resulting in the final 3D framework of **1**. Furthermore, complex **1** can be excited directly and emit strong solid-state fluorescence at room temperature, which may make them excellent candidates for fluorescent materials.

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